TI-871

6/23/53

WADC TECHNICAL REPORT 52-291

AD012965

AN INVESTIGATION OF VARIOUS PROPERTIES OF NiA1

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SEPTEMBER 1952

WRIGHT AIR DEVELOPMENT CENTER

NOTICES

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AN INVESTIGATION OF VARIOUS PROPERTIES OF NiAl

Richard L. Wachtell
American Electro Metal Corporation

September 1952

Materials Laboratory
Contract No. AF 33(038)-10716
RDO No. 615-17

Wright Air Development Center
Air Research and Development Command
United States Air Force
Wright-Patterson Air Force Base, Ohio

McGregor & Werner, Inc., Dayton, O.
300 - June, 1953

FOREWORD

This report was prepared by the American Electro Metal Corporation at the completion of work on Contract No. AF 33(038)-10716, Supplemental Agreement 2 (52-2252). The project began as a study of the Aluminides of Molybdenum (AFTR 6601, Parts 1 and 2), and has been continued under the supplemental agreement cited above as a study of the aluminides of nickel.

Work on this project was initiated under Research and Development Order No. 615-17, "Ceramic Materials", and was administered under the direction of the Materials Laboratory, Directorate of Research, Wright Air Development Center, with Mr. L. D. Richardson acting as project engineer for the Air Force, while the technical work has been done at the laboratories and by the staff of the American Electro Metal Corporation. Project leader was R. Wachtell, with W. Herz assisting. The work has been under the general supervision of G. Stern, Vice-President and Technical Director of the American Electro Metal Corporation.

ABSTRACT

Production of the alloy NiAl and a modified composition NiAl + 5% Ni has proved feasible as well as its subsequent fabrication by powder metallurgical technique. Properly hot-pressed bars of the NiAl + 5% Ni composition show strengths in modulus of rupture as high as 144,000 psi at room temperature and 68,000 at 980° C.

The air oxidation resistance of the modified NiAl + 5% Ni composition is excellent up to 1095° C., with weight gains of the order of 1.25 MG./CM^2 being exhibited after 300 hours of exposure.

Heat shock properties are likewise excellent, as judged by NACA tests, and by performance in the Air Force Heat Shock apparatus.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDING GENERAL:

M. E. SORTE
Colonel, USAF
Chief, Materials Laboratory
Directorate of Research

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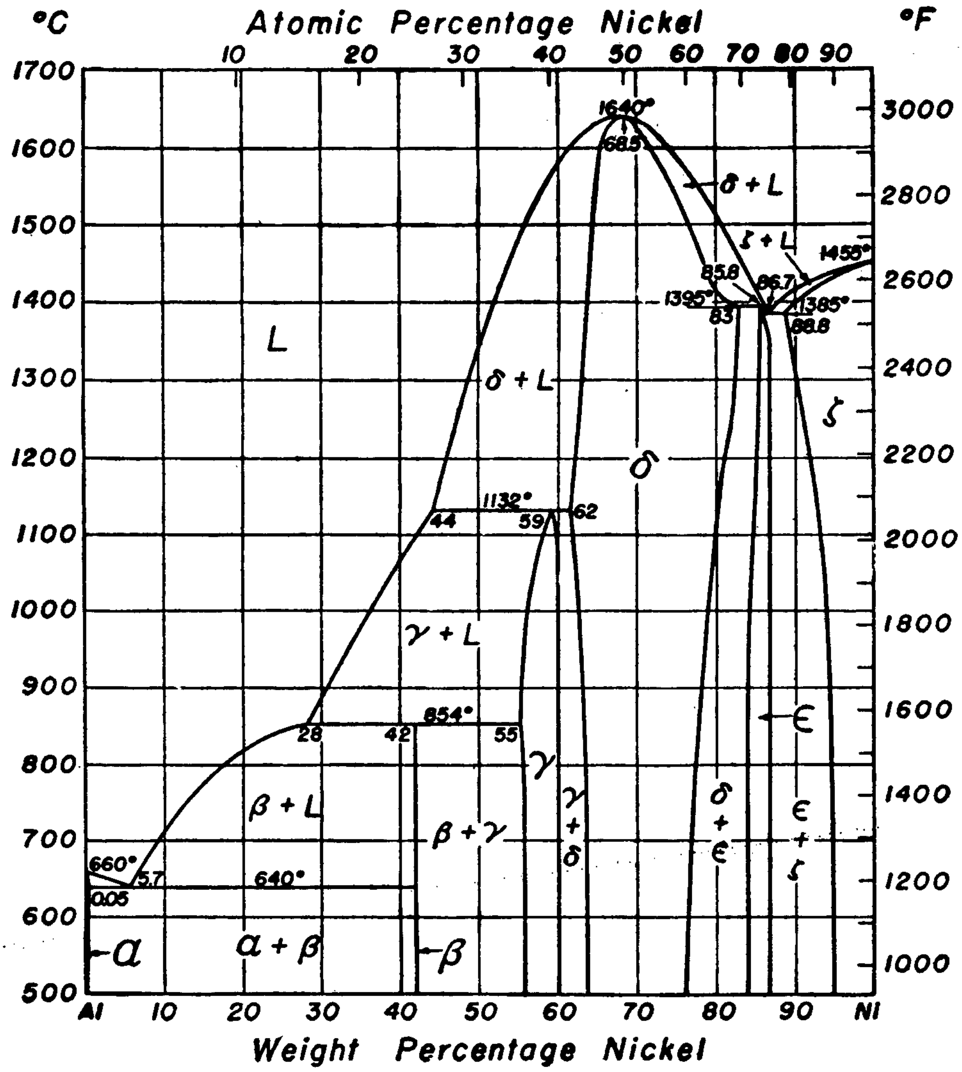
INTRODUCTION

Considerable work has recently been completed in determining the physical properties of the system Mo-Al,[1] with a view to determining what, if any, value this material might have as a refractory. The data obtained have been sufficiently interesting to stimulate a further study of systems with aluminum, with the same general ends in view.

A consideration of the phase diagrams of these systems, as listed in the Metals Handbook, shows a number of such systems which suggest investigation because of the high melting point of certain of their compounds, and preliminary studies have been made of alloys of aluminum with Zr, Ti, Cr, W and Ni. Based upon these studies, one of the most hopeful of these systems appears to be the aluminum-nickel system. An intermetallic, NiAl, is known to have a high melting point (about 1650° C., well above the melting point of Ni itself). The diagram has been well established, and the system is fairly simple (Fig. 1)[2]. In addition, nickel, while not in free supply, has domestic sources. Work has therefore been started on the system aluminum-nickel, and this report details the progress made during the work on this project.

Al-Ni Aluminum-Nickel

By W. L. Fink* and L. A. Willey*



The diagram shown is from investigations by Alexander and Vaughan,[2] Schramm,[3] and Fink and Willey.[1]

The Curie temperature of nickel is decreased linearly from 360 to 70 C by the addition of 4.6% Al in solid solution.[3]

The solubility of nickel in solid aluminum is, as follows:[1] 0.040% at 625 C, 0.018 at 570, 0.006 at 505. The solubility of aluminum in solid nickel is:[3] 9.7% at 1300 C, 8.0 at 1200, 6.5 at 1080, 6.0 at 980, 5.0 at 700.

Nickel is used as an alloying element in a few commercial aluminum-base alloys, principally in aluminum-copper-magnesium alloys and aluminum-silicon-magnesium alloys. Nickel increases the strength of these alloys at elevated temperature and decreases the coefficient of thermal expansion, but usually decreases the resistance to corrosion.

At the other end of the system there are a few commercial alloys that are used principally for thermocouple wires.

References

1 W. L. Fink and L. A. Willey, Trans AIME, 111, 293 (1934)

2 W. O. Alexander and N. B. Vaughan, J Inst Metals, 61, 247 (1937)

3 J. Schramm, Z Metallkunde, 33, 347 (1941)

4 A. J. Bradley and A. Taylor, Proc Roy Soc (London), A 159, 56 (1937)

* Aluminum Research Laboratories, New Kensington, Pa.

Crystal Structures and Lattice Parameters of Aluminum-Nickel Alloys

Phase	Crystal Structure	Lattice Parameter, kX units a	b	c
β	Orthorhombic	6.598	7.352	4.802
γ	Trigonal	4.028	4.891	....
δ	Body-centered cubic	2.881	....	....
ε	Face-centered cubic	3.58	....	....

FIG. 1 Phase Diagram, The System Aluminum/Nickel

I - POWDER MANUFACTURING

Nickel-aluminum alloy powders have been prepared in the laboratory by direct synthesis of the two parent metals. Both carbonyl nickel and nickel from the Metals Disintegrating Company, Incorporated, (comminuted, melted electrolytic nickel) were used as nickel sources. The aluminum used was M. D. atomized Al. Laboratory analyses were as follows:

99.3 Ni	99.3 Al
0.25 C	0.3 Fe
0.33 Fe	0.2 S
	0.05 Cu
	0.04 Mg
	0.1 Al_2O_3

Synthesis of the Ni-Al alloys was accomplished by mixing the powders and pressing into slugs. The slugs were stacked into a magnesia crucible and the whole placed in a covered graphite receptor and heated by high frequency. Argon or hydrogen was used as an atmosphere throughout this synthesis.

The nickel and aluminum react at about 650° C. to form an alloy. This reaction while not explosive is nevertheless accompanied by a large evolution of heat. A 1 kg mass of powder evolves enough heat to fuse itself, i.e., exceed 1650° C. This strongly exothermic reaction has been observed before in working with casting of nickel-aluminum alloys.[3) 4)] Initial syntheses have been made of alloys of 68% Ni composition, since this represents the highest melting composition of the system.

The fused slug of metal obtained after synthesis is quite porous. A substantial evolution of gas appears to accompany the synthesis process. Despite this, the slug is quite tough and a drop hammer is needed for initial size reduction in the powder comminuting cycle. Following drop hammer, the chips are fed to a jaw crusher, and then ball milled to final size in a carbide mill using tungsten carbide slugs. This mill is completely lined with tungsten carbide and its inside dimensions are 4" diameter and 8" height. Here the toughness of the material again becomes apparent, and ball milling times of three days are needed to reduce the powder to -325 mesh size. Ball milling is done in ethyl alcohol. Following milling, the powder is separated by filtering and is then vacuum dried in a Eimer and Amend vacuum desiccator.

II - METHODS OF MANUFACTURING BODIES

Three methods of making bodies have been investigated. These have been: (1) hot pressing; (2) cold pressing and sintering; and (3) pre-hot pressing and subsequent sintering. The first of these has proven most successful to date.

Hot pressing is done in the conventional manner using graphite dies, resistance heated. Temperature and pressure are reached within about one minute. Outside die temperature may range from 1325-1425° C., but time at temperature must be extended at the lower temperatures if dense pieces are to be obtained, and curtailed at the upper end if burning is to be avoided. The effect of pressure has not been determined.

There is no tendency for the material to stick to the graphite, and pieces pressed hot from the die usually fall clear of the punches spontaneously.

Cold pressing and sintering has proved difficult. Laminated bars are the rule rather than the exception. The sintering temperatures needed are of the order of 1500-1600° C., and have been achieved so far by the use of Lindberg Globar heated laboratory furnaces fitted with special tubes (Morganite, triangle H-5, impervious mullite).

The high percentage of aluminum in these alloys makes them sensitive to oxidation, and atmosphere troubles have been experienced in sintering. To date, the best atmosphere found has been Argon gettered by molybdenum aluminide in powdered form. Better success in sintering has been achieved with the use of small slugs pre-pressed at 1200° C. in the hot press. These densify and shrink and are free from laminations which are most troublesome in cold pressed bars.

III - TESTING OF NiAl COMPOSITIONS

The testing of NiAl compositions has been conducted with the background thought that a refractory material is being sought. Tests to date have included:

(1) Oxidation test in air.

(2) Oxidation test in kerosene burner.

(3) Room temperature modulus of rupture.

(4) Elevated temperature modulus of rupture.

(5) Heat shock testing (NACA test and Air Force deflection vane test)

(6) Stress to rupture.

(7) Resistance to fuming HNO_3.

(8) Electrical resistivity.

(9) Thermal expansion.

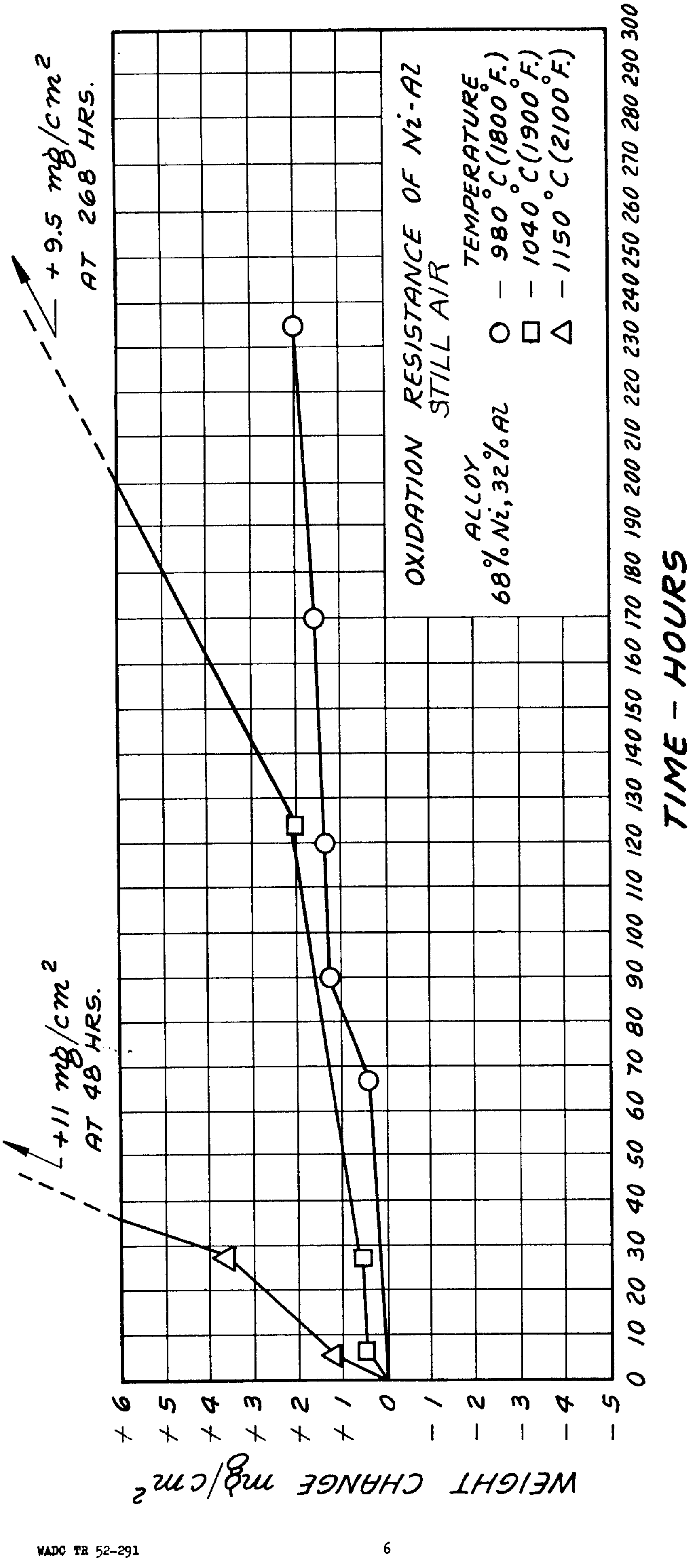
(10) Microstructure of NiAl.

The test results may be summarized as follows:

(1) Oxidation Testing in Air[1)]

Oxidation testing began with specimens hot pressed from the 68% Ni balance Al (nominal) composition. The tests were conducted in still air in open ended quartz tubes in Lindberg laboratory furnaces. The specimens were supported on palladium plates. As will be seen from Figure 2, performance was satisfactory at 980° C., and at 1040° C., but at 1150° C. the weight gain was considered excessive.

Following earlier experience gained on studies of Mo-Al alloys, the basic 68% Ni alloy powder was modified by additions of 5% Ni and 5% Al ball milled in immediately before hot pressing. The



OXIDATION RESISTANCE OF Ni-Al
STILL AIR
ALLOY
68% Ni, 32% Al
TEMPERATURE
○ – 980°C (1800°F.)
□ – 1040°C (1900°F.)
△ – 1150°C (2100°F.)
+11 mg/cm²
AT 48 HRS.
+9.5 mg/cm²
AT 268 HRS.
WEIGHT CHANGE mg/cm²
+6
+5
+4
+3
+2
+1
0
–1
–2
–3
–4
–5
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
210
220
230
240
250
260
270
280
290
300
TIME - HOURS

FIG. 2

effect of these additions is shown in Figures 3 and 4. It is apparent that the additional Al is harmful, and the additional Ni helpful. Both mixtures still remain within the NiAl region on the phase diagram, but the additional nickel may be considered the more desirable modification since the nickel-rich side shows a much more gradual drop in temperature for formation of a liquid phase (see Figure 1). Thus a small percentage of excess Al will cause a liquid phase to form at about 1125° C., whilst up to 75% Ni the alloys still melt above 1500° C.

An oxidation curve for a pre-alloy of composition equivalent to that of the 68% Ni, 32% Al, modified with 5% Ni before pressing, has been obtained and is shown in Figure 5. This curve does not fully express the differences, however, since the pre-alloy forms an oxide which is flaky and easily falls from the compact. In laboratory weighing this oxide is disturbed as little as possible. Under service conditions, the tenacious oxide film of the modified alloy would be more desirable.

Oxidation tests were also carried out at lower temperatures, in order to be sure that no anomaly exists, such as was encountered with molybdenum aluminides.[1)] Results are reported in Figures 6, 7 and 8. As will be seen, only a slight change is noted as the temperature is decreased to 650° C.

Each curve in Figures 2 to 7 is representative for one specimen which was measured after various times at the indicated temperature.

(2) Oxidation Test in Kerosene Burner

Because of the intended application of the material under development, it has for some time been contended that air oxidation

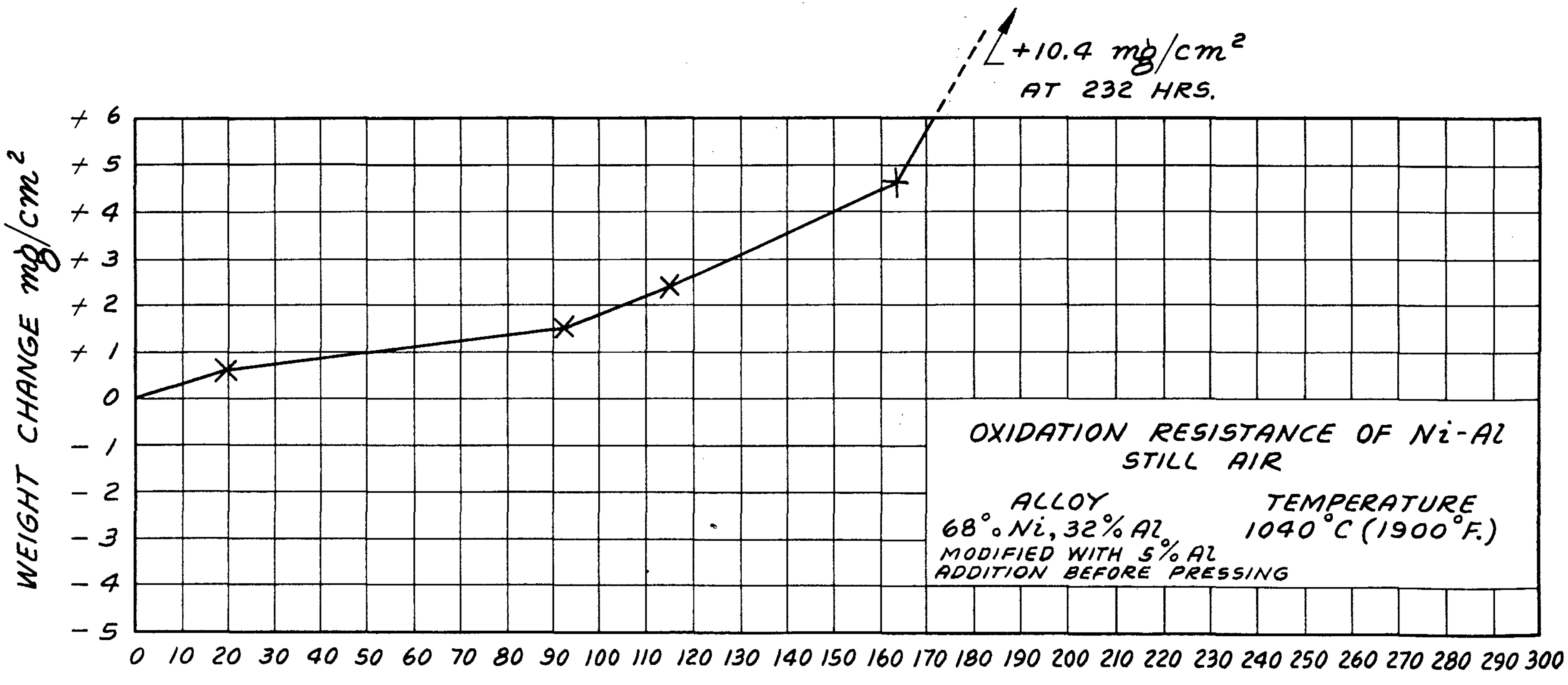
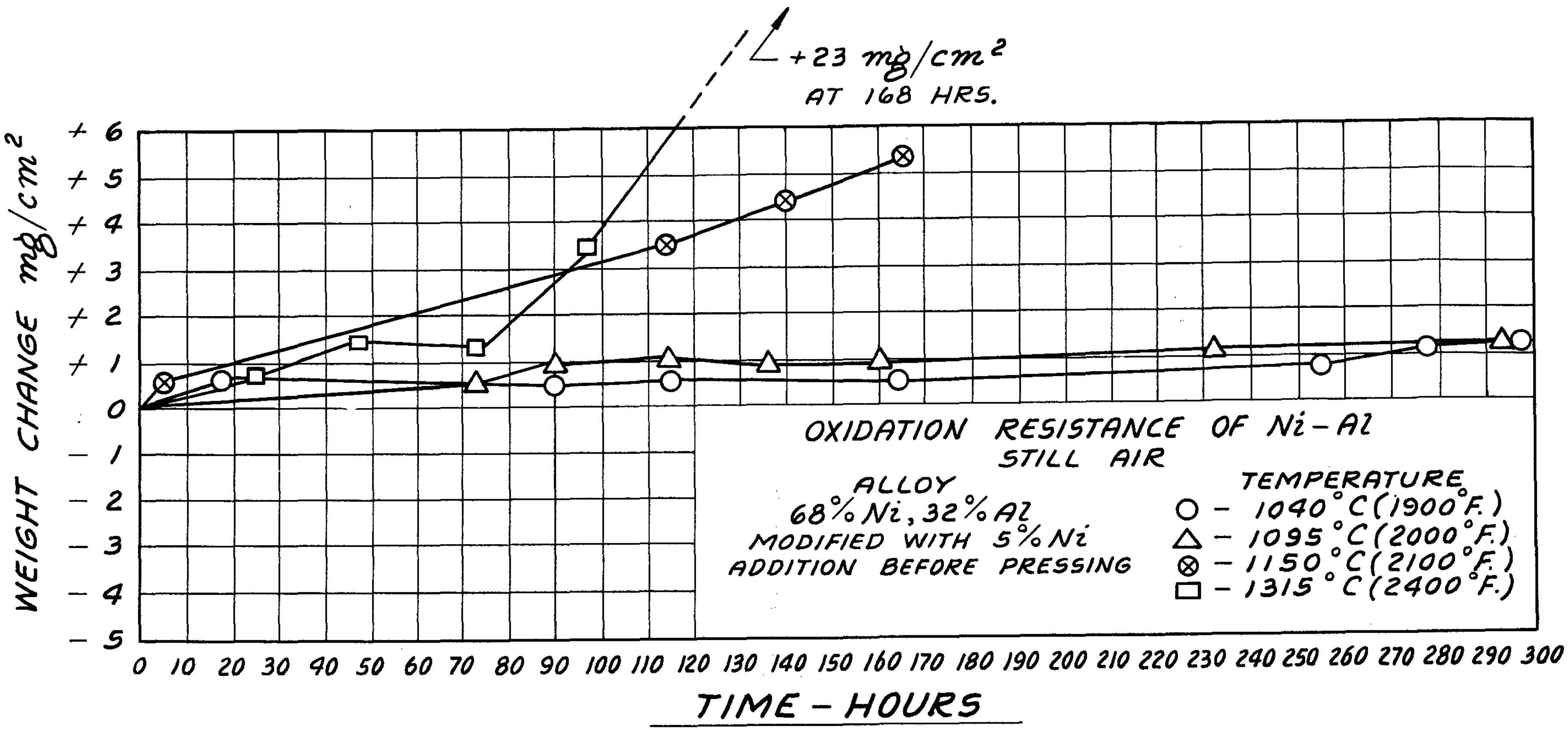

+10.4 mg/cm²
AT 232 HRS.
OXIDATION RESISTANCE OF Ni-Al
STILL AIR
ALLOY
68% Ni, 32% Al
MODIFIED WITH 5% Al
ADDITION BEFORE PRESSING
TEMPERATURE
1040°C (1900°F.)
WEIGHT CHANGE mg/cm²
+6
+5
+4
+3
+2
+1
0
-1
-2
-3
-4
-5
0 10 20 30 40 50 60 70 80 90 100 110 120 130 140 150 160 170 180 190 200 210 220 230 240 250 260 270 280 290 300
TIME - HOURS

FIG. 3



+23 mg/cm²
AT 168 HRS.
OXIDATION RESISTANCE OF Ni-Al
STILL AIR
ALLOY
68% Ni, 32% Al
MODIFIED WITH 5% Ni
ADDITION BEFORE PRESSING
TEMPERATURE
○ - 1040 °C (1900 °F.)
△ - 1095 °C (2000 °F.)
⊗ - 1150 °C (2100 °F.)
□ - 1315 °C (2400 °F.)
WEIGHT CHANGE mg/cm²
+6
+5
+4
+3
+2
+1
0
-1
-2
-3
-4
-5
0 10 20 30 40 50 60 70 80 90 100 110 120 130 140 150 160 170 180 190 200 210 220 230 240 250 260 270 280 290 300
TIME - HOURS

FIG. 4

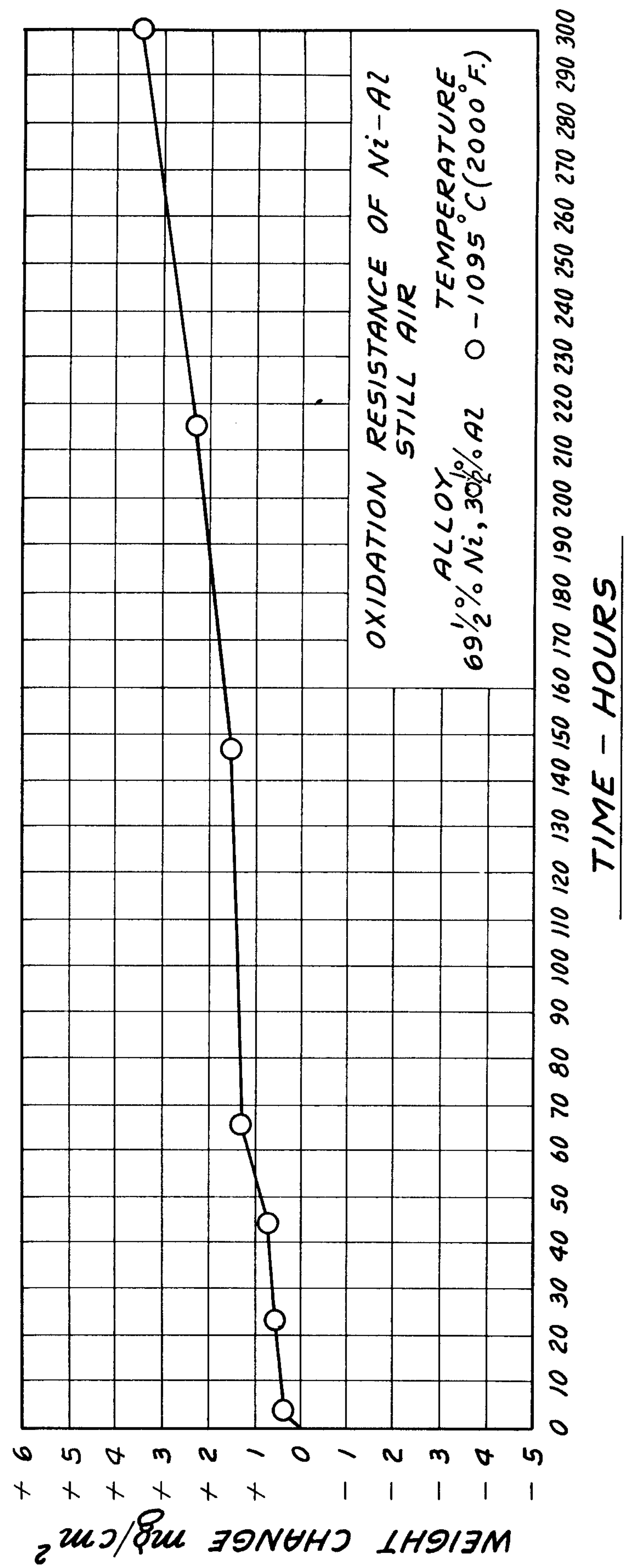

OXIDATION RESISTANCE OF Ni-Al
STILL AIR
ALLOY
69½% Ni, 30½% Al
TEMPERATURE
○ - 1095°C (2000°F.)
TIME - HOURS
WEIGHT CHANGE mg/cm²
0 10 20 30 40 50 60 70 80 90 100 110 120 130 140 150 160 170 180 190 200 210 220 230 240 250 260 270 280 290 300
+6 +5 +4 +3 +2 +1 0 -1 -2 -3 -4 -5

FIG. 5

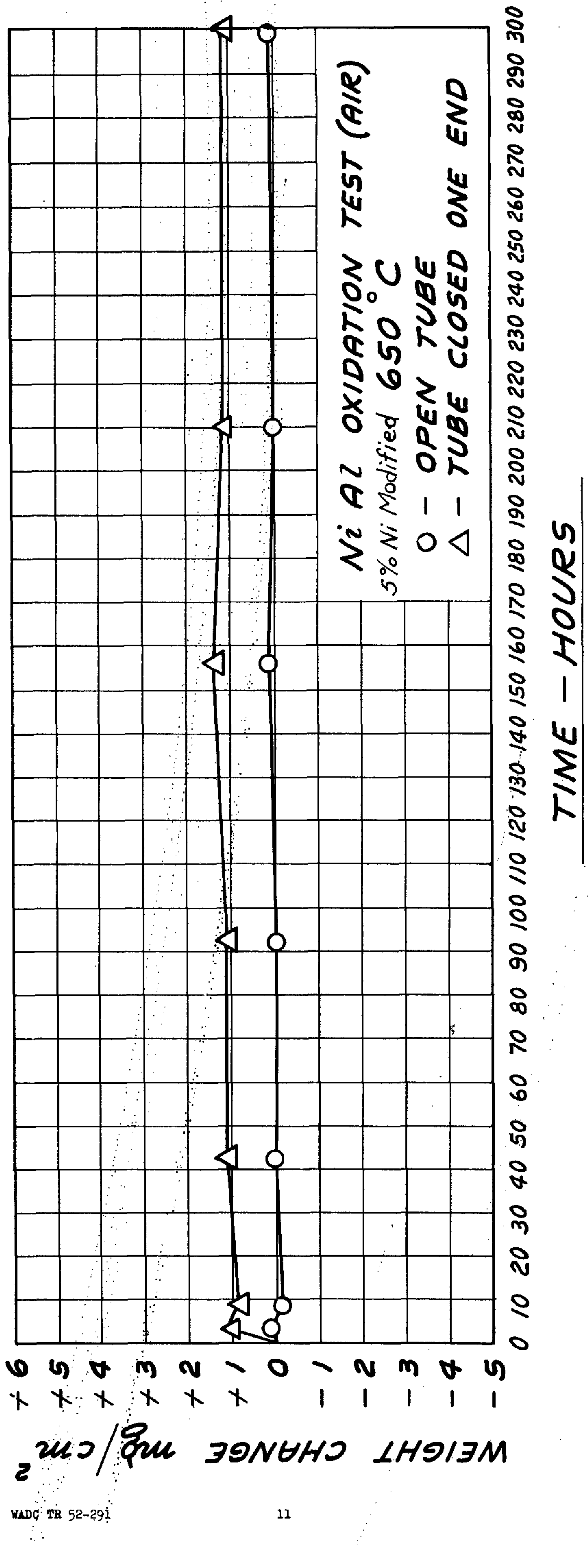
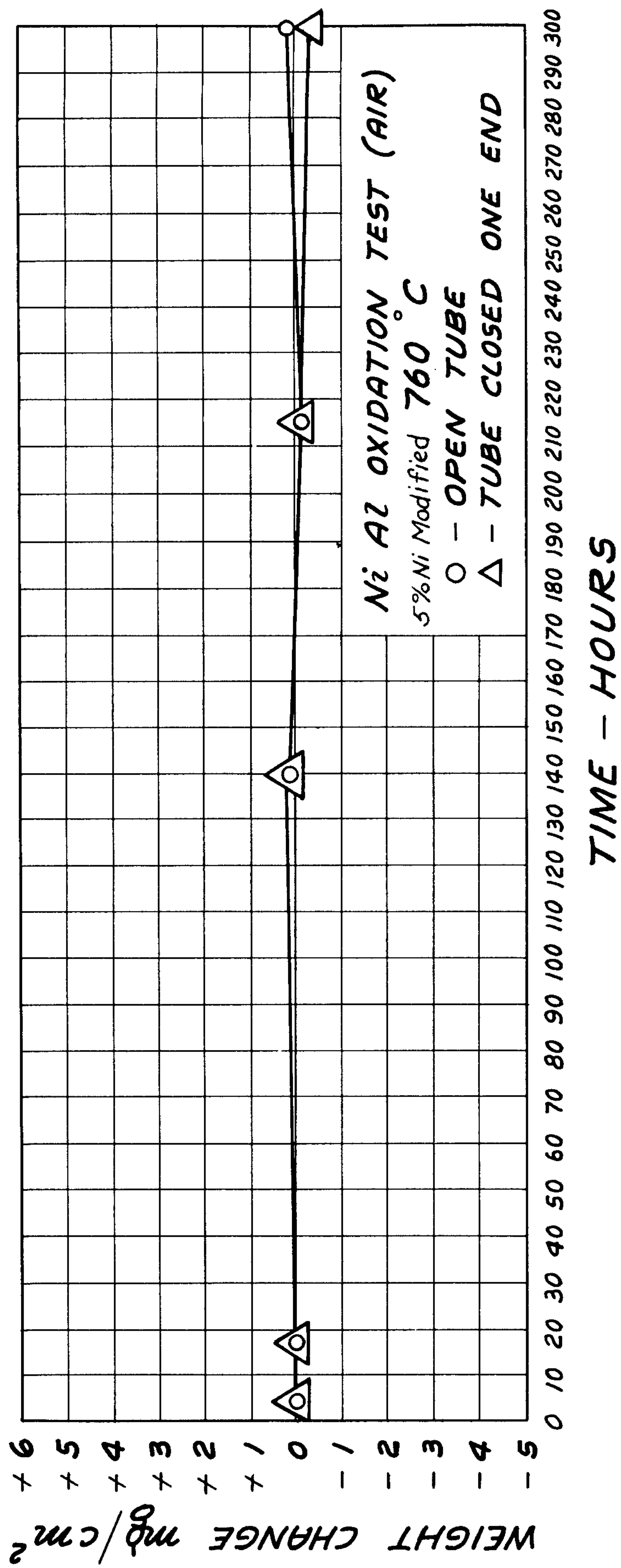
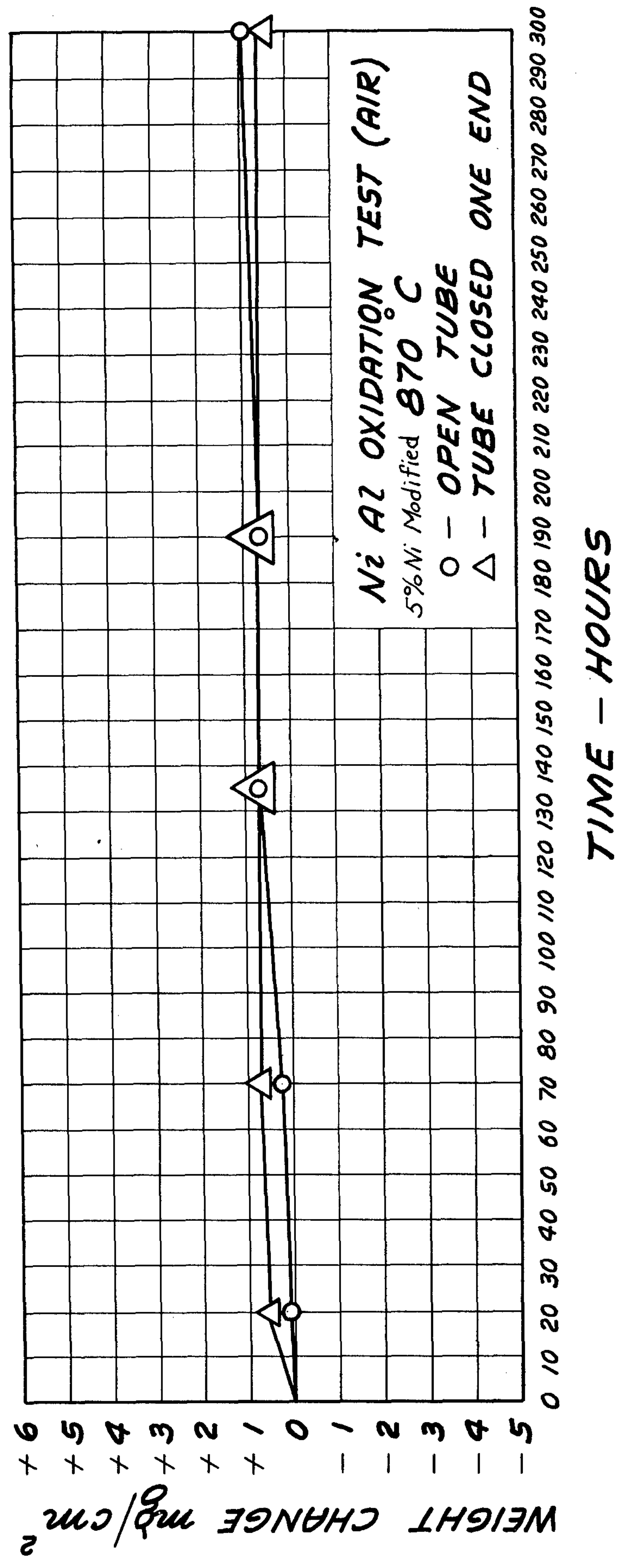

Ni Al OXIDATION TEST (AIR)
5% Ni Modified 650° C
O – OPEN TUBE
△ – TUBE CLOSED ONE END
TIME – HOURS
WEIGHT CHANGE mg/cm²
0 10 20 30 40 50 60 70 80 90 100 110 120 130 140 150 160 170 180 190 200 210 220 230 240 250 260 270 280 290 300
+6 +5 +4 +3 +2 +1 0 -1 -2 -3 -4 -5

FIG. 6



Ni Al OXIDATION TEST (AIR)
5%Ni Modified 760 °C
○ - OPEN TUBE
△ - TUBE CLOSED ONE END
TIME - HOURS
WEIGHT CHANGE mg/cm²
+ 6
+ 5
+ 4
+ 3
+ 2
+ 1
0
- 1
- 2
- 3
- 4
- 5
0 10 20 30 40 50 60 70 80 90 100 110 120 130 140 150 160 170 180 190 200 210 220 230 240 250 260 270 280 290 300

FIG. 7



Ni Al OXIDATION TEST (AIR)
5% Ni Modified 870°C
○ - OPEN TUBE
△ - TUBE CLOSED ONE END
TIME - HOURS
WEIGHT CHANGE mg/cm²
0 10 20 30 40 50 60 70 80 90 100 110 120 130 140 150 160 170 180 190 200 210 220 230 240 250 260 270 280 290 300
+6 +5 +4 +3 +2 +1 0 -1 -2 -3 -4 -5

FIG. 8

testing gives only a partial picture of the usefulness of the alloys. It must be recognized that materials which perform quite well in an electrically heated furnace passing a few cubic feet of air may perform very poorly when exposed to combustion gases from petroleum products with enormously rapid atmosphere changes.

For this reason an apparatus has been designed and built, which will permit a closer approximation to service conditions in turbine engines than does an electric tube furnace, or even a kerosene or oil-fired furnace.

Essentially, the apparatus consists of a wheel, to the periphery of which are attached the test specimens, and which is whirled while under the impinging flame of two compressed air-kerosene burners. An enclosing furnace, bearings, drive, and associated control equipment constitute the entire apparatus which is illustrated in Figures 9 - 14.

The furnace has been designed to accommodate specimens which are a slight modification of the standard modulus bar (Figure 15) and it is intended to determine what strength and other changes occur as oxidation progresses. The maximum test temperature will be 2000° F.

No conclusive results have been obtained with this equipment at the time of this report.

(3) Room Temperature Modulus of Rupture[1)]

The room temperature modulus of rupture of the NiAl + 5% Ni material under study has proven good. Maximum values of 144,000 psi have been obtained and, with present knowledge and techniques, better than 100,000 can be consistently obtained even on long (2-1/2") modulus bars. It is interesting to note that a definite though slight yield point appears to exist in these materials. Just before failure, a

1 - KEROSENE BURNER NOZZLES
2 - KEROSENE STORAGE TANK
3 - THERMOCOUPLE ACCESS AND SIGHT HOLES
4 - END BEARING
5 - TEST SPECIMEN ROTOR WHEEL
6 - COOLING FINS
7 - VARIABLE SPEED DRIVE AND MAIN BEARING ASSEMBLY
8 - ROLLERS FOR ACCESS TO TEST CHAMBER
9 - TEMPERATURE CONTROLS AND SAFETY CUT-OUT

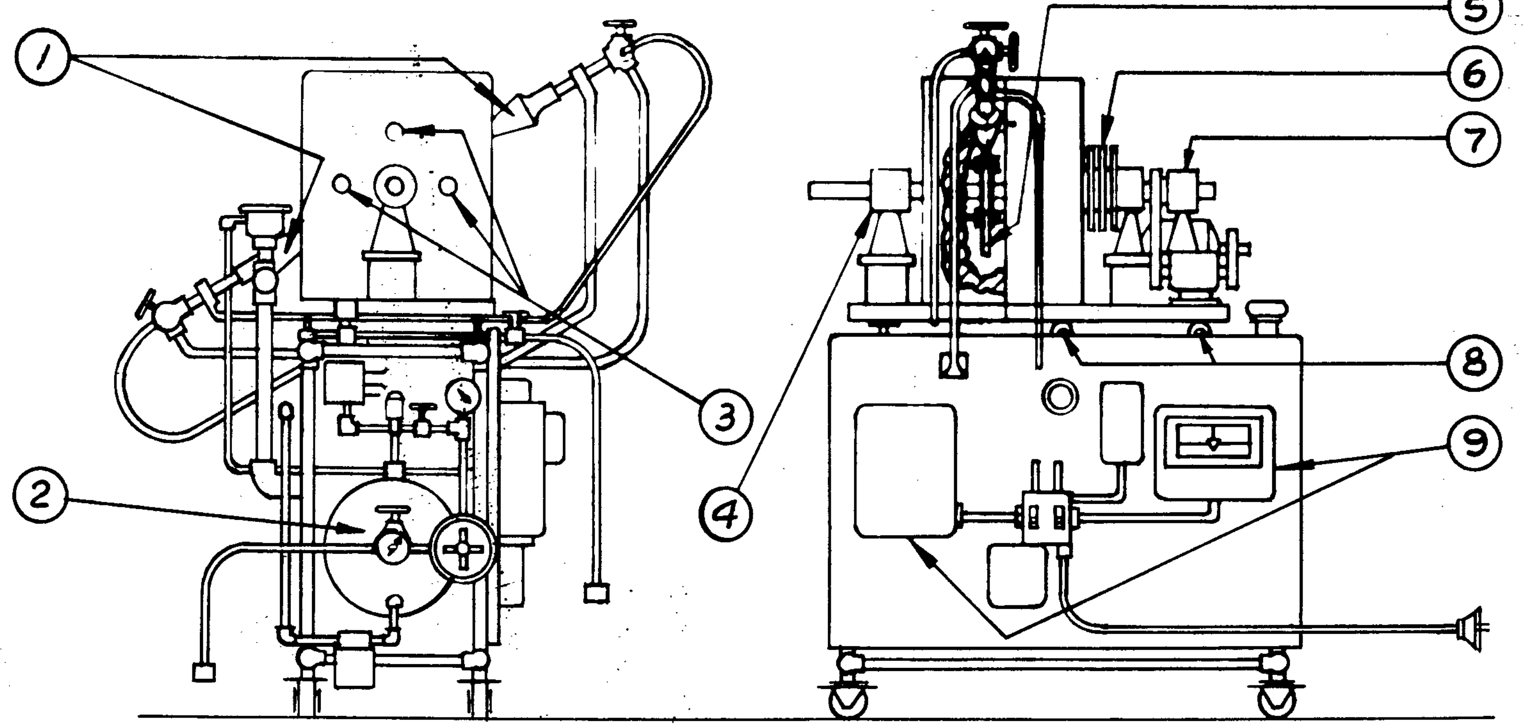


FIG. 9

KEROSENE BURNER OXIDATION TESTING DEVICE

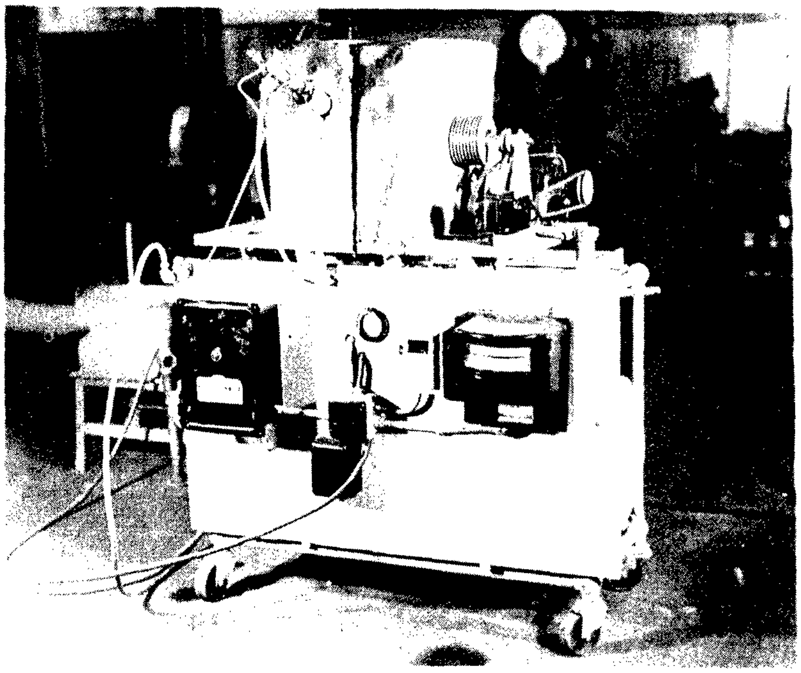


FIG. 10. General view of kerosene burner oxidation apparatus, showing temperature and safety controls, drive for rotor, and one of the two burner nozzles.

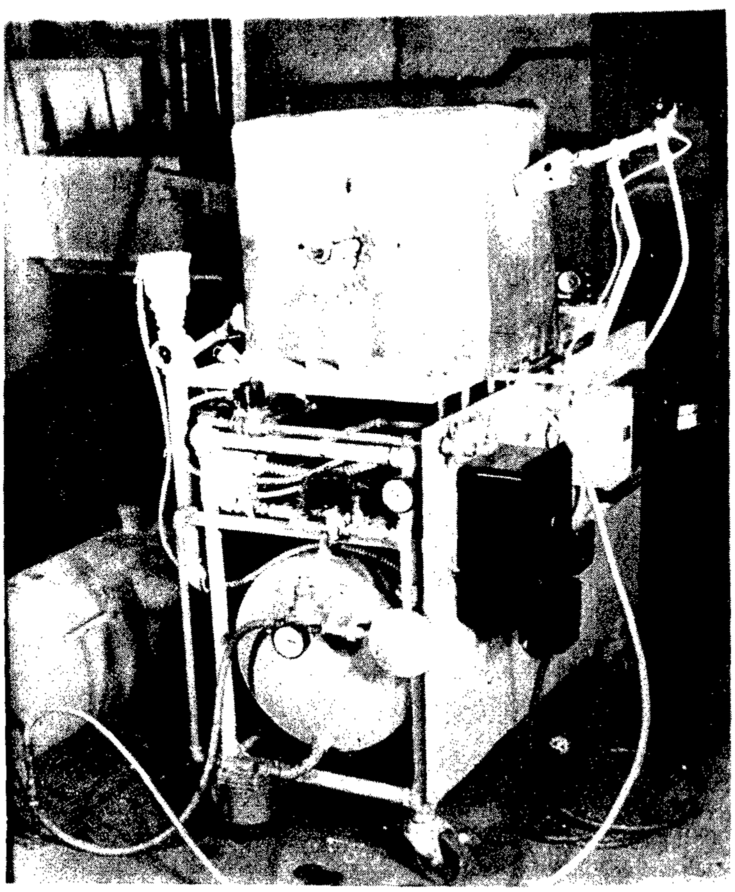


FIG. 11. End view of kerosene burner oxidation apparatus, showing kerosene and air piping, solenoids, pressure regulators, and kerosene storage tank.

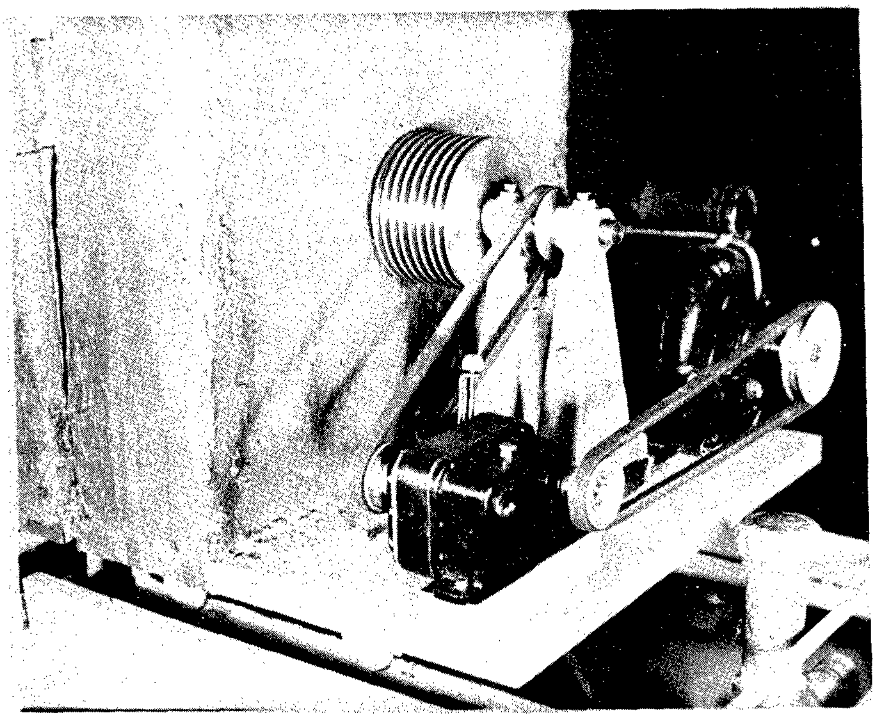


FIG. 12. Detail of rotor drive; motor, variable speed drive, main bearing, and rotor shaft with pressed-on aluminum cooling fins.

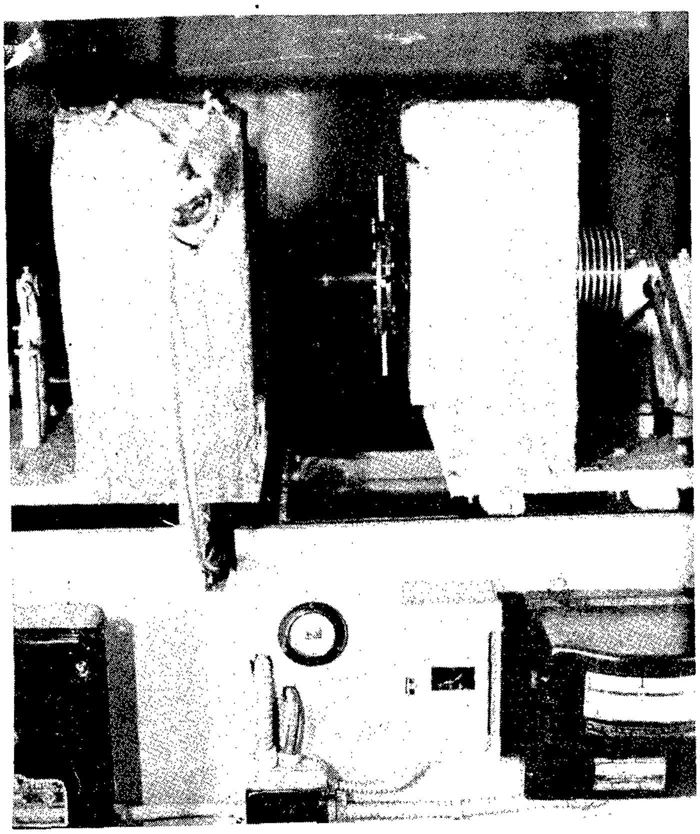
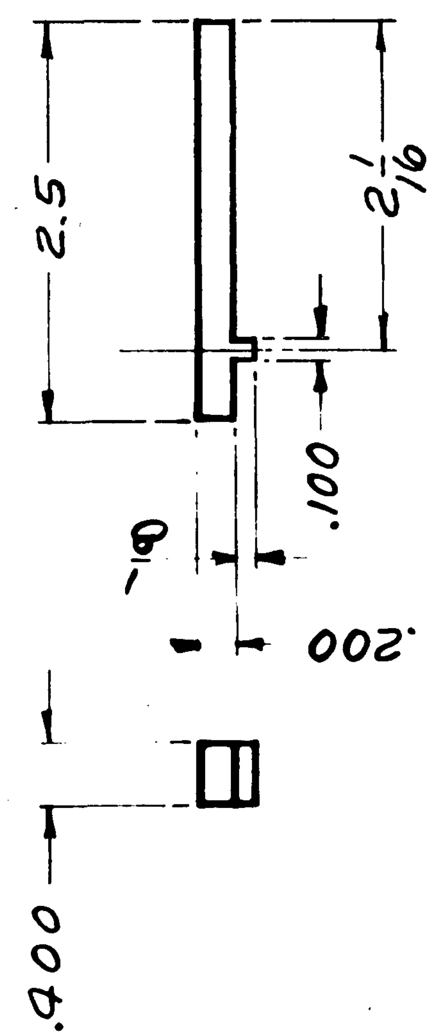


FIG. 13. Detail showing drive section rolled back on tracks exposing rotor and mounted specimens for inspection, insertion, or removal. The round object lower center is a running time meter.

FIG. 14. Detail showing method of mounting specimens. The retaining ring is bolted to the grooved rotor with stainless bolts. The specimen shape has since been modified to that shown in Fig. 15 to provide better support and clearance.



MODIFIED MODULUS OF RUPTURE SPECIMEN
FOR USE IN KEROSENE BURNER TEST

FIG. 15

distinct "drop of beam" is noted in the testing machine, although this appears as a momentary halt rather than a drop. This is, of course, suggestive of some ductility in the material, but no quantitative data could be obtained. Table I lists modulus values for materials produced in several ways.

TABLE I

Physical Properties of Hot Pressed and Pre-Hot Pressed and Sintered NiAl

Composition	Processing Temp., °C.	Time At Heat (sec)	Densities	Rockwell A	Modulus of Rupture (Room Temp.)	Remarks
68 Ni + 32 Al + 5 Ni	1400	90	6.00	68	130,000	
	1400	90	6.10	68	130,000	
	1400	90	6.07	68	134,000	
	1400	90	6.00	68	127,000	
	1400	90	6.05	68	114,000	Hot Pressed
	1400	90	5.95	68	128,000	
	1400	90	6.09	68	123,000	
	1400	90	6.09	68	137,000	
	1400	90	6.10	68	139,000	
68 Ni + 32 Al + 5 Ni	1600	720	5.90	70	95,000	Pre-Hot pressed at 1200° C. and sintered in H atmosphere
	1500	720	5.70	70	81,000	
	1500	720	5.82	70	81,000	
	1600	720	5.80	68	81,000	
69.5 Ni + 32 Al	1400	90	6.12	70-71	105,000	Hot Pressed
	1400	90	6.13	72.5	114,000	
	1400	90	6.00	70.3	102,000	

(4) Elevated Temperature Modulus of Rupture[1)]

Tests of modulus conducted at 950 and 980° C. have developed values of about one-half the room temperature properties. These tests were first conducted at a time when best practice developed only 50,000 to 60,000 psi at room temperature, and subsequently on stronger specimens which can be produced at this time, with the same ratio holding.

Too few tests have been made so far to report quantitative data.

(5) Heat Shock Testing (NACA Test,[5] Air Force Deflection Vane Test)[6]

Tests have been conducted on standard NACA discs through two full cycles: i.e., 25 cycles at 1800, 2000, 2200, 2400° F., (980, 1090, 1200, 1315° C., approx.), and then repeat; without failure. In addition (as discussed in Section IV) two Air Force deflection vanes have passed 1000 cycles of test without failure.

(6) Stress to Rupture

Stress to rupture testing has been started on specimens hot pressed to the shape shown in Figure 16. The first test conducted at 816° C. (1500° F.) with a load of 10,000 psi, failed at 250 hours. Not too much confidence is placed in this result, and a more extensive test program is being planned.

(7) Resistance to Fuming HNO_3

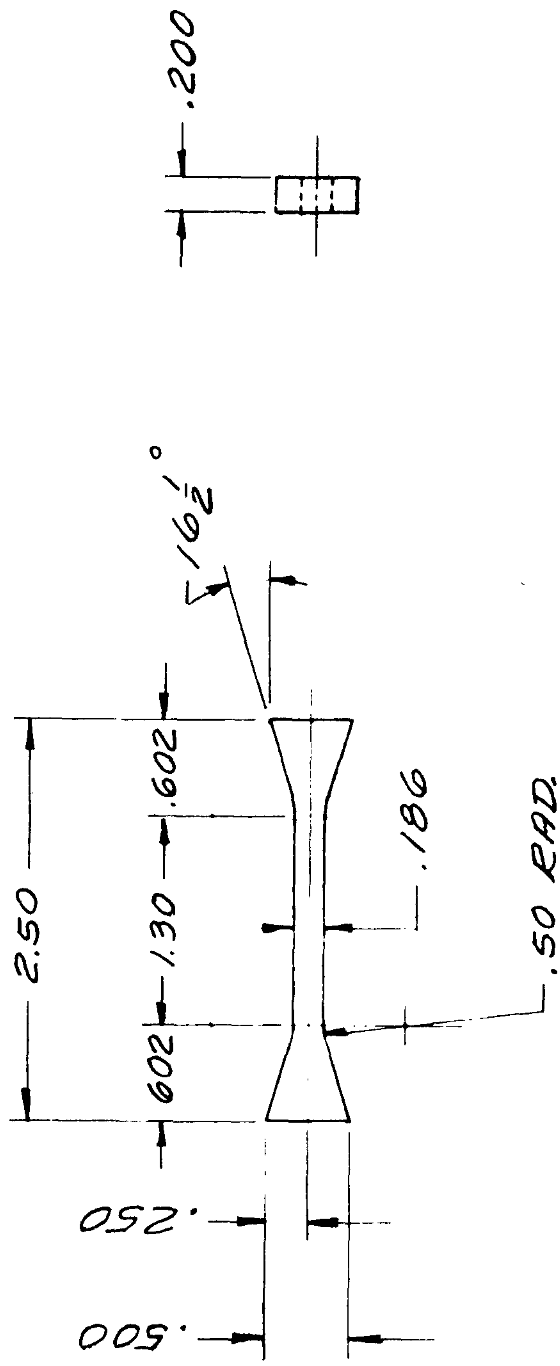
Resistance to red and white fuming HNO_3 has been determined at room temperature. The samples was immersed into the acid and weighed at various time intervals after careful drying. The acids used were:

white fuming nitric acid:
Reagent Grade Merck, Sp. G. 1.5

red fuming nitric acid:
Reagent Grade Baker & Adamson, Sp. G. 1.59 to 1.6

Figure 17 shows the data developed.

In addition, tests at Bell Aircraft have been started, with that Company reporting "negligible" attack after 30 days at room temperature and "negligible" attack after 15 days at 71° C. (160° F.). Extrapolated from data gathered after 30 days, a corrosion rate of 2.65×10^{-2}



STRESS RUPTURE SPECIMEN

FIG. 16

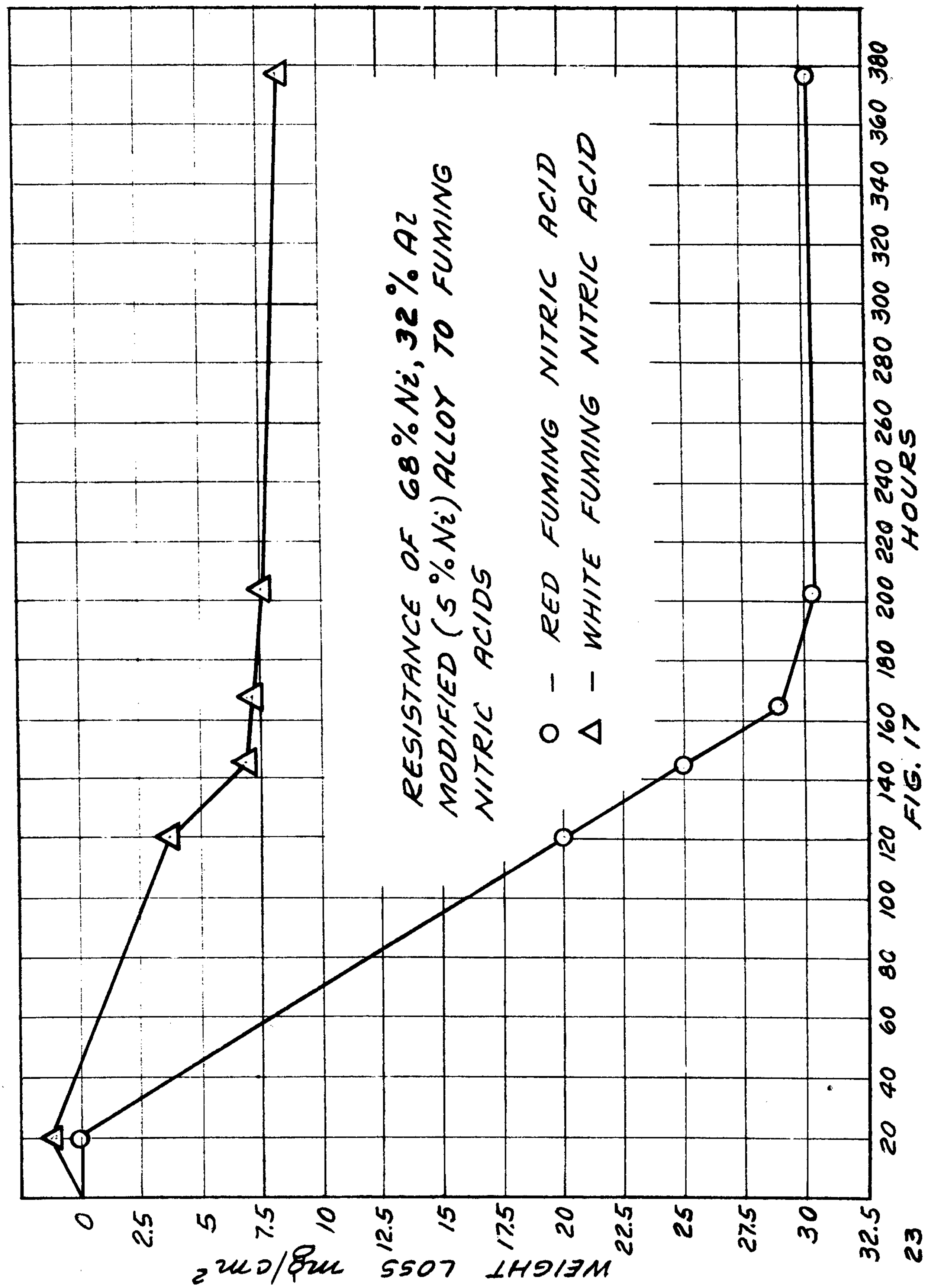

RESISTANCE OF 68% Ni, 32% Al MODIFIED (5% Ni) ALLOY TO FUMING NITRIC ACIDS
○ – RED FUMING NITRIC ACID
△ – WHITE FUMING NITRIC ACID
HOURS
20 40 60 80 100 120 140 160 180 200 220 240 260 280 300 320 340 360 380
WEIGHT LOSS mg/cm²
0 2.5 5 7.5 10 12.5 15 17.5 20 22.5 25 27.5 30 32.5

FIG. 17

23

inches per year is reported for the higher temperature test.

(8) Electrical Resistivity[1)]

The electrical resistivity has been determined on a few samples chiefly to establish an order of magnitude. The values obtained are 20-30 microhm centimeters, which classifies the materials as definitely metallic, and probably with metallic thermal conductivity properties.

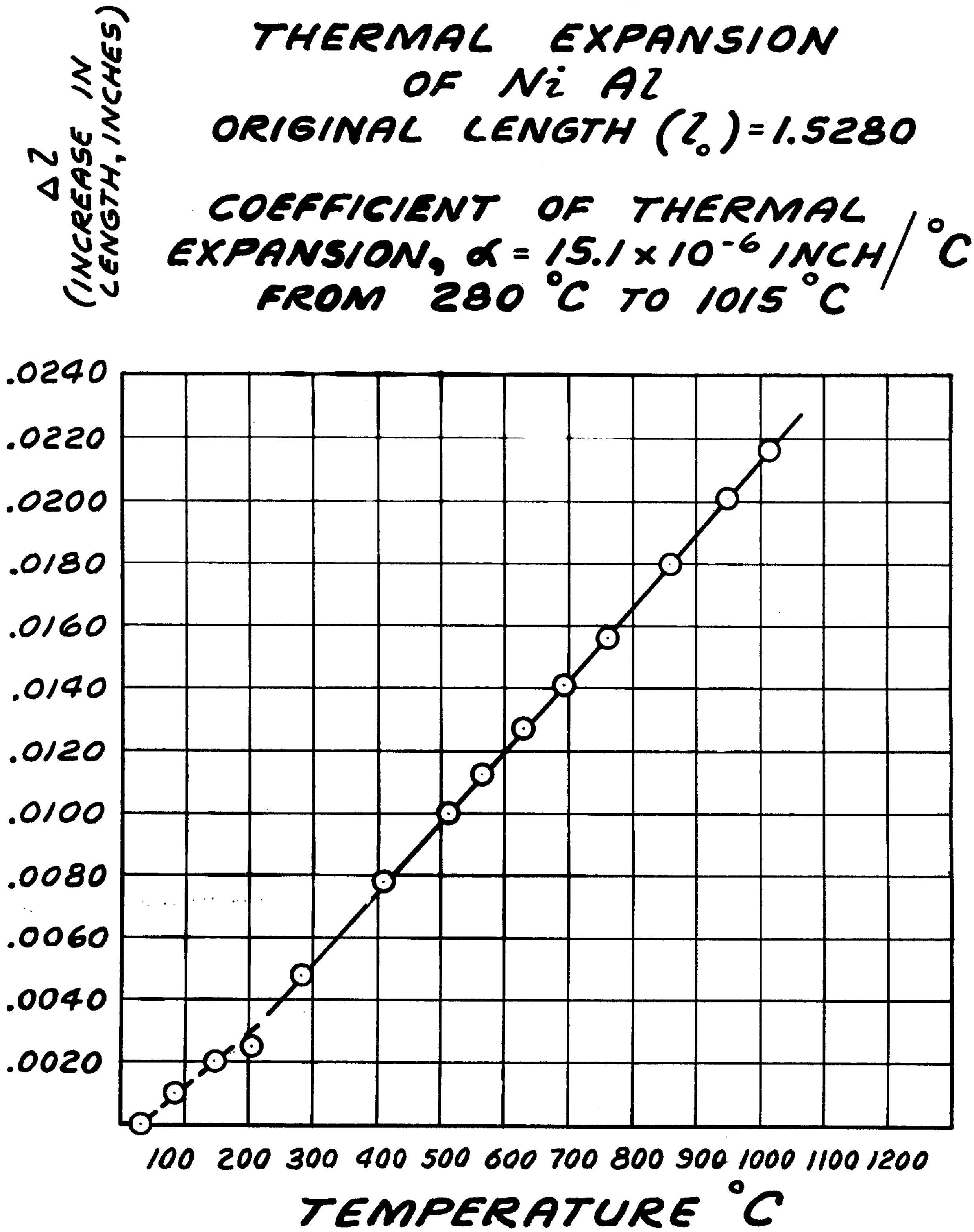
(9) Coefficient of Thermal Expansion

Tests have been conducted to determine the coefficient of thermal expansion of hot pressed NiAl. The measurements were made in a dilatometer with a quarts feeler rod and a dial indicator which could be read to 0.0001". The furnace is heated by Nichrome wire, and its maximum temperature is about 1100° C. The value obtained is 15.1×10^{-6} inch/inch/°C. between 280 and 1015° C. The curve reproduced in Figure 18 shows the actual data obtained.

(10) Microstructure of the Nickel Aluminides

The results of some micrographic examinations of NiAl are given in Figures 19, 20. The grain size is remarkably unaffected by long periods at heat during oxidation testing, possibly because of oxides incorporated by the manufacturing method. All properly hot pressed specimens are quite dense. The dark areas visible in the photomicrograph are not voids, but oxide particles.

The etchant used consisted of 1 part of HCl and 3-5 parts of ethyl alcohol. The etching was done electrolytically at 1 volt and 0.5 amps. for 10 sec.



THERMAL EXPANSION
OF Ni Al
ORIGINAL LENGTH (l_o) = 1.5280
COEFFICIENT OF THERMAL EXPANSION, $\alpha = 15.1 \times 10^{-6}$ INCH/°C
FROM 280 °C TO 1015 °C
Δl (INCREASE IN LENGTH, INCHES)
.0240
.0220
.0200
.0180
.0160
.0140
.0120
.0100
.0080
.0060
.0040
.0020
100 200 300 400 500 600 700 800 900 1000 1100 1200
TEMPERATURE °C

FIG. 18

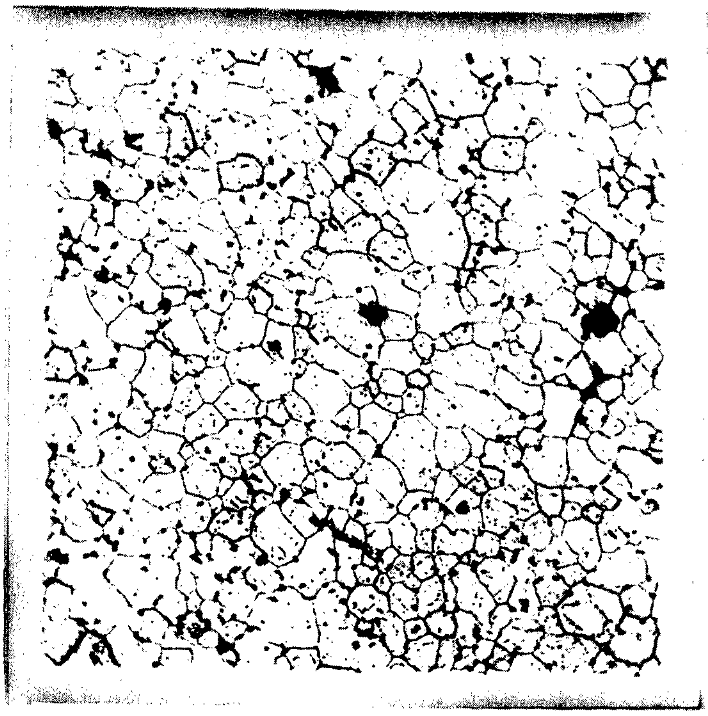


FIG. 19. NiAl x 250
Normal structure as hot pressed; equiaxed grains.

Etchant:
HCl (conc.)- 1 part
Ethyl Alcohol- 3-5 parts
Etch electrolytically

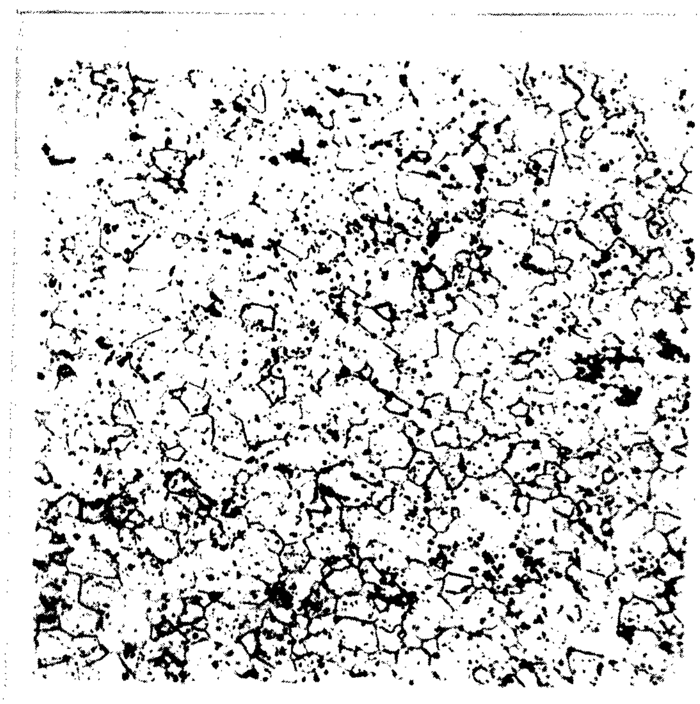


FIG. 20. NiAl x 250
NiAl hot pressed specimen after 170 hrs. at 1360°C. Etched as above.

IV - PARTS MANUFACTURE

(1) Deflection Vanes

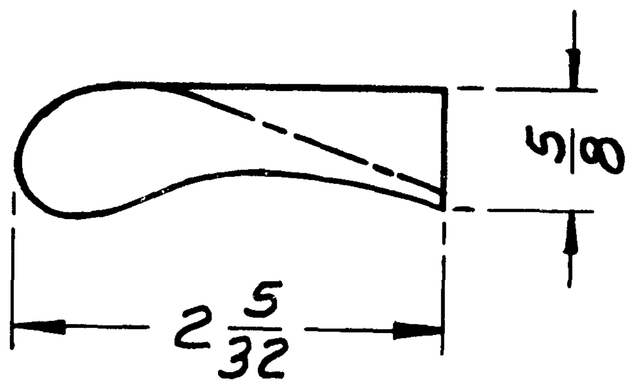
A consideration of the heat shock and oxidation resistance properties of the NiAl material developed has suggested that it be tested in the Air Force heat shock apparatus,[6)] which utilizes an engine deflection vane as the test specimen. Considerable effort has therefore been devoted to the manufacture of engine deflection vanes.

Five kilograms of NiAl powder were prepared for these experiments in order to allow for preparation of all parts from a single lot of powder, and to allow for a large anticipated wastage.

Only hot pressing was considered as a possible method of manufacture, since sintering, even by the best known present practice, did not produce parts of equal strength.

Two different procedures were planned in hot pressing. In the first, a body was to be pressed with the undercamber to final shape, but with the uppercamber omitted on the trailing portion of the vane, past the point of maximum thickness. Figure 21 shows the cross-section of this shape. It was intended, by this means, to avoid the extreme variation in section thickness between trailing edge and mid-line of the vane, which creates a serious fill problem. Subsequently, the vane was to be machined (diamond ground) to final shape as shown by the dotted line, Figure 21.

Because of the considerable inherent waste of material in this approach, attempts were also to be made to press the blade to final shape, leaving only a small amount of "oversize" for cleaning up.



HOT PRESS PRE-SHAPE, TO BE GROUND INTO AIR FORCE DEFLECTION VANE

FIG. 21

After several unsuccessful tries, parts were produced by both methods (Figure 22). The best procedure appeared to be to use somewhat low pressing temperatures (1325-1350° C., outside die wall) and extended holding times (10-12 minutes). This minimized the danger of overheating and grain growth or burning in the part. It should be mentioned that the interior of the die is undoubtedly much hotter than the 1350° C. mentioned above. The precise temperature has not been measured.

Six blade shapes were produced, of which, after grinding and radiographic inspection, three were adjudged sound. These were forwarded to the contracting authority for testing. At the time of present writing, two of these three blades have been tested, both passing through the entire 1,000 cycle series without heat shock failure and without important oxidation.

(2) Forging

As part of a general consideration of methods of manufacture of parts, attempts have been made to "hot-coin" or "forge" blanks of NiAl + 5% Ni which had been first pre-hot pressed. The forging hammer used was a 300 lb. Chambersberg board hammer, fitted with dies closed on the ends, but open at the sides. The forged pieces were square in cross section, about 1/2" on a side, and about 3" long. The temperature used in forging was about 1280°-1300° C., a limit imposed by the available furnace. It is believe that higher temperatures would be more desirable.

Forging was accomplished with surprising success. There was definite upset and flow of metal in the die, a coherent flash was

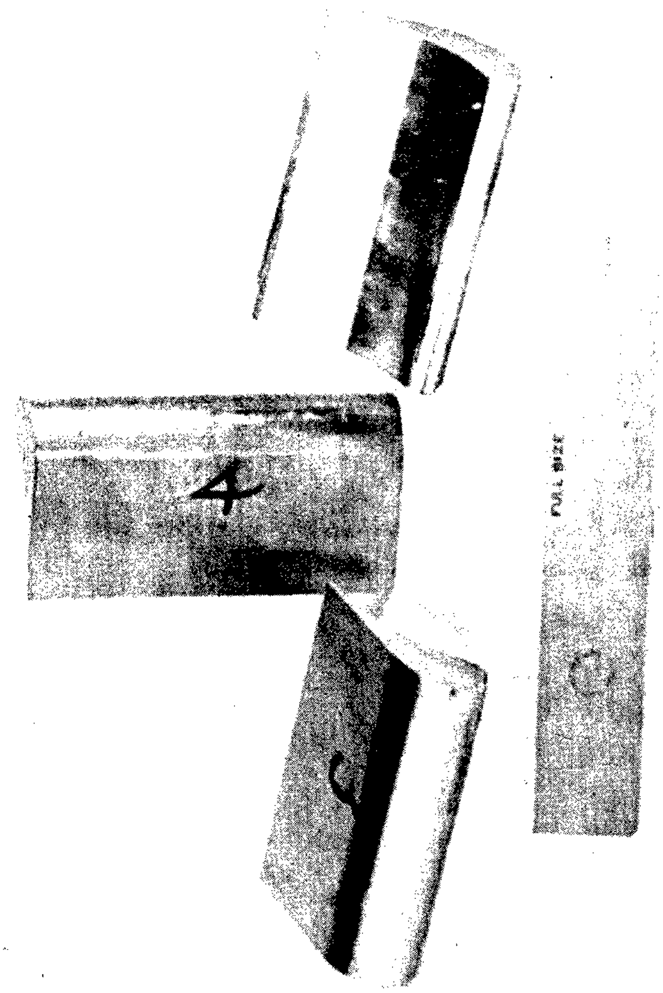


FIG. 22 Air Force Deflection Vanes for Heat Shock Testing

formed, flow lines were observed in the microstructure (Figure 23, compare with Figure 19) and sound bars were obtained. It seems not impossible that deflection vanes, buckets, etc. can be made by a forging technique from NiAl + 5% Ni.

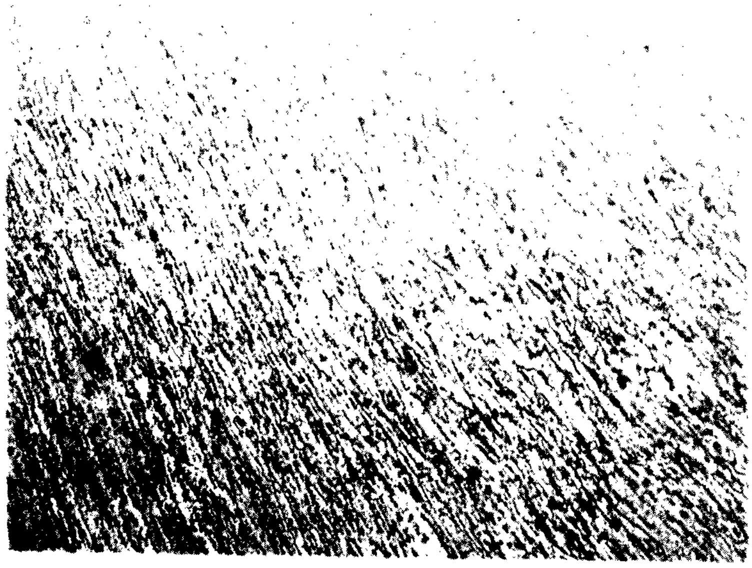


FIG. 23. NiAl x 250

Structure as forged showing flow lines and fibrous structure developed. Compare with Fig. 19.

V - MODIFICATION OF THE ALLOY

Because of the extremely high nickel content (approx. 70%) in the material as developed, thought has been given to reducing the requirement for critical material in the alloy. As a first step, iron has been substituted for part of the nickel. Following the same powder manufacture procedure as for Ni Al, an alloy has been compounded having the composition 30 Ni 40 Fe 30 Al.

In first experiments, this material developed a modulus of rupture of 68,000 psi (max.) and oxidation resistance as indicated in Figure 24.

This material is definitely worth further experimental study.

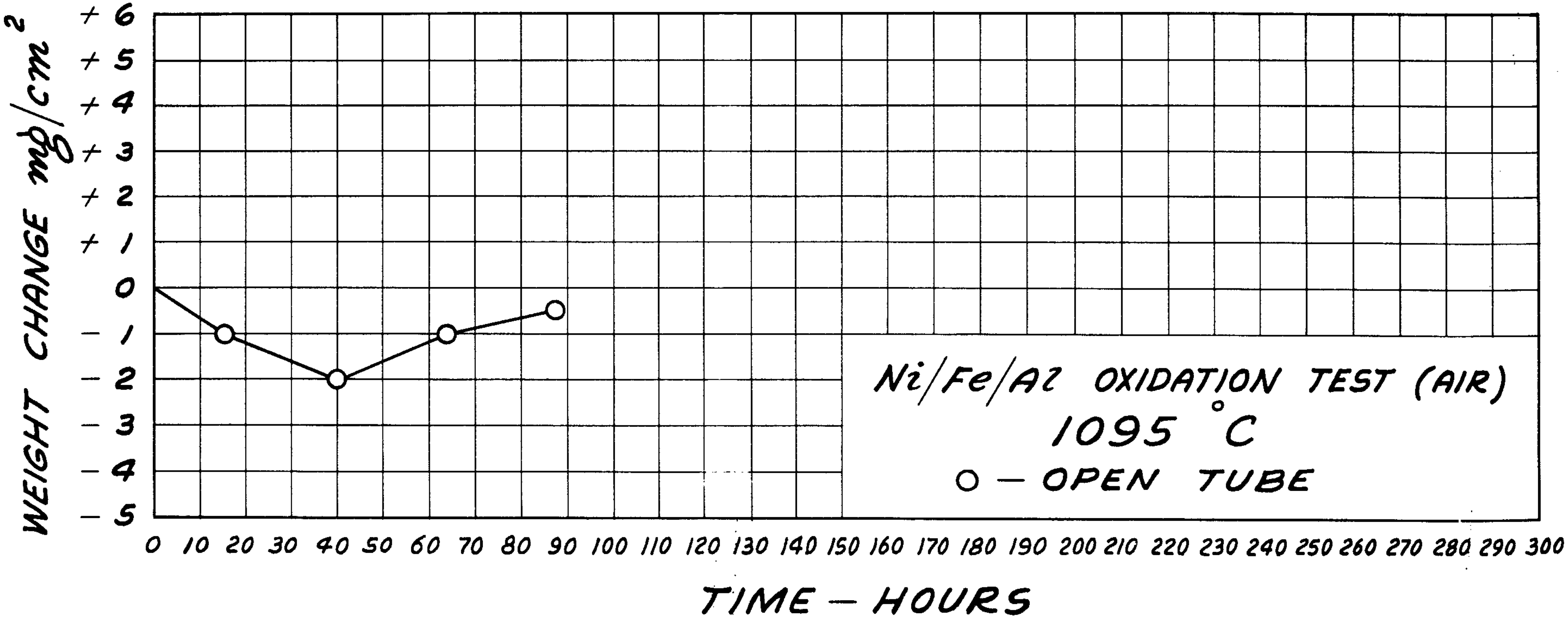

WEIGHT CHANGE mg/cm²
+ 6
+ 5
+ 4
+ 3
+ 2
+ 1
0
− 1
− 2
− 3
− 4
− 5
0 10 20 30 40 50 60 70 80 90 100 110 120 130 140 150 160 170 180 190 200 210 220 230 240 250 260 270 280 290 300
Ni/Fe/Al OXIDATION TEST (AIR)
1095 °C
O − OPEN TUBE
TIME − HOURS

FIG. 24

VI - SUMMARY AND CONCLUSIONS

(1) An alloy of the composition NiAl + 5% Ni has been produced and bodies fabricated therefrom by powder metallurgical methods.

(2) It has been established that excellent air oxidation resistance attaches to properly made alloys of this approximate composition up to temperatures of 1090° C. (2000° F.), with weight gains of the order of 1.25 MG/CM^2 being exhibited after 300 hours of exposure.

(3) Maximum room temperature modulus of rupture values of 144,000 psi have been attained with such materials at the present state of the art, and indications of some ductility before fracture have been noted.

(4) Heat shock tests indicate excellent properties for this material.

(5) Resistance to red and white fuming nitric acid has been determined at room temperature, with good resistance found.

(6) Resistivity values of 20-30 microhm cm. have been established as approximate values, indicating metallic behavior.

(7) A coefficient of thermal expansion of 15.1×10^{-6} inch/inch °C. has been found for the material.

(8) Air Force heat shock vanes have been successfully manufactured and tested.

(9) Indications have been found that the alloy can be successfully forged.

VII - REFERENCES

1) "An Investigation of the Alloys of Aluminum and Molybdenum", R. L. Wachtell, Air Force Technical Report #6601, U. S. Air Force, Wright Air Development Center, Wright Patterson Air Force Base, Ohio.

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4) AG. C. Gwyer, 2. Anorg. Chem. 57, 113, 1908.

5) NACA RM No. E9B07, Correlation of Physical Properties of Ceramic Materials with Resistance to Fracture by Thermal Shock by W. G. Lidman and A. R. Bobrowsky, April 19, 1949

6) Memorandum Reports MCREXP-506-398 and MCREXP-506-321, Power Plant Laboratory, Directorate of Laboratories, W.A.D.C.